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(See Section 6)

MILITARY SPECIFICATION

PHOSPHATE COATINGS, HEAVY, MANGANESE OR ZINC BASE (FOR FERROUS METALS)

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers two types of heavy phosphate coating for ferrous metals, applied by immersion. The coatings consist of a manganese phosphate or zinc phosphate base and, when specified, a supplementary treatment (see 6.1).

1.2 Classification. Coatings shall be of the following types and classes, as specified (see 6.2):

Type M	Manganese phosphate base
Class 1	Supplementary preservative treatment or coating, as specified
Class 2	Supplementary treatment with lubricating oil conforming to MIL-L-3150
Class 3	No supplementary treatment
Class 4	Chemically converted (may be dyed to color as specified). With no supplementary coating or supplementary coating as specified

Beneficial comments (recommendations, additions, deletions), and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization Division (611), Indian Head, MN 20640, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

Type Z	Zinc phosphate base
Class 1	Supplementary preservative treatment or coating, as specified
Class 2	Supplementary treatment with preservative conforming to MIL-C-16173, Grade 1 or MIL-L-3150 (as alternate for very small parts)
Class 3	No supplementary treatment
Class 4	Chemically converted (may be dyed to color as specified). With no supplementary coating or supplementary coating as specified

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

TT-C-490	Cleaning Methods and Pretreatment of Ferrous Surfaces for Organic Coatings

MILITARY

MIL-P-116	Preservation-Packaging, Methods of
MIL-L-3150	Lubricating Oil, Preservative, Medium
MIL-C-16173	Corrosion Preventive Compound, Solvent Cutback, Cold-Application
MIL-P-50002	Phosphate Coating Compounds, For Phosphating Ferrous Metal

* STANDARDS

MILITARY

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-1218	ACS Chemicals

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

AMERICAN PUBLIC HEALTH ASSOCIATION AND AMERICAN WATERWORKS ASSOCIATION

Standard Methods for the Examination of Water and Waste Water, 13th Edition, 1971

(Applications for copies should be addressed to the American Public Health Association, 1015 18th Street, NW, Washington, DC 20036, or the American Waterworks Association, 2 Park Avenue, New York, NY 10016).

AMERICAN SOCIETY FOR TESTING AID MATERIALS (ASTM)

ASTM E 8	Tension Testing of Metallic Materials
ASTM B 117	Salt Spray (Fog) Testing

(Applications for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103).

AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI)

B46.1 - 1962	Surface Texture

(Applications for copies should be addressed to the American National Standards Institute, Inc., 1430 Broadway, New York, NY 10018).

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

3. REQUIREMENTS

* 3.1 Preproduction inspection. Unless otherwise specified (see 6.2), a preproduction inspection is required and shall be used to verify that the process complies with 3.2. Details of the procedure, chemicals, and the equipment proposed to he used by the contractor shall be submitted in writing via the contracting officer to the procuring activity,

and written approval shall be received from the contracting officer prior to production. The exact designation of all materials proposed for use, together with the names of the manufacturers, shall be stated. The proposed procedure shall include a detailed method of control, with limits for time, temperature, pH values, and all other pertinent details that will ensure compliance with the requirements of this specification. No deviation from the approved process shall be permitted without prior written approval of the procuring activity. Approval of process, materials and equipment implies no guarantee of acceptance of the results obtained in use. Regardless of the processes or materials approved, the phosphate coatings shall conform to all the applicable requirements of this specification.

3.2 Process. The phosphate coatings shall be applied under the controls and in the stages described below. A single contractor shall perform all phases of the coating process. Unless otherwise specified (see 6.2), phosphate coatings shall be applied after all machining, forming, welding, and stress relieving heat treatment have been completed, but prior to hydrogen embrittlement relief treatment.

3.2.1 Bath materials and controls. The bath materials and controls of the bath shall be in accordance with the following:

* a. The materials used in the baths shall be capable of producing phosphate coatings conforming to all requirements herein for the type and class specified. Government activities shall use materials in accordance with MIL-P-50002 (see 6.4.1).

b. The bath concentration, temperature, and immersion time for parts shall be such that the phosphate coating meets all the requirements of this specification.

c. The equipment shall be constructed of materials resistant to the action of the phosphating solutions and free from copper alloy fittings or brazing in contact with the solution.

d. Phosphatized parts shall not be allowed to dry between the phosphatizing solution treatment and the subsequent cold running water rinse. Fog sprays or other means for prevention of drying shall be provided.

* e. The supplier of the phosphate coated parts shall maintain a permanent record (see 3.2.1g) of the history of each processing bath, showing all additions of chemicals to the bath, and the results of all analyses performed.

f. Control of the chemical content of the phosphate coating baths shall consist of determination of free acid, total acid, and ferrous

iron. Unless otherwise specified (see 6.2), the frequency of testing for chemical content shall be determined by the user but must be sufficiently frequent to assure adequate control so that it can be demonstrated that no lot of parts is processed when the bath is not within the established limits. Analysis for free and total acid should be made just prior to the processing of each lot or every two hours, whichever is less frequent. The ferrous iron content should be analyzed just prior to the processing of each lot or weekly, whichever is less frequent.

g. Permanent records of bath controls shall be maintained for not less than one year (seven when specified (see 6.2)) and made available to the Government upon request. Permanent records shall be defined as legible records printed in ink or typed in lieu of pencilled notes.

* 3.2.2 Stage 1, cleaning. Cleaning shall be in accordance with method I, II, III, or IV of TT-C-490, or a combination thereof under the following conditions. Abrasive blasting is the preferred treatment prior to heavy phosphating and shall be utilized unless otherwise specified and/or approved by the procuring activity (see 6.2) for reasons of dimensional control or surface finish roughness control that make it necessary to omit abrasive blasting (see 3.2.2.1.d and 6.5).

3.2.2.1 Rust or scale removal. Rust or scale shall first be removed by dry, abrasive blasting using sand or steel grit in accordance with method I of TT-C-490 under the following conditions:

* a. Parts shall be free of oil, grease, dirt, and other contamination or else degreased before abrasive blasting.

b. Abrasive tumbling shall not be used.

c. After blasting, any abrasive residue shall be removed by a blast of dry, compressed air.

* d. Abrasive blasting shall be controlled and performed using a grit size sufficiently fine to preserve the required RMS surface finish roughness. Where abrasive blasting will damage the finish, method III, V or VI of TT-C-490 may be used to remove light rust provided they do not adversely affect the coating quality. Strong acids or alkaline solutions shall not be used prior to manganese base phosphate coating without subsequent surface conditioning to refine the coating grain and remove the effects of such solutions on the surface reactivity and the phosphate coating process (see 6.5).

3.2.2.2 Alkaline or emulsion cleaning. When method III or IV of TT-C-490 is used, cleaning shall be followed by a thorough rinsing in clean water.

3.2.3 Stage 2, phosphating. The phosphate coating shall be applied by immersion under the following conditions:

* a. Type M coatings shall be manganese base and type Z shall be zinc base. All materials used for coatings shall be approved by the procuring activity as specified in 3.1 or shall be in accordance with MIL-P-50002.

b. The bath concentrations, temperature, and immersion time for parts shall be such that the phosphate coating meets all the requirements of this specification.

* 3.2.4 Stage 3, water rinse. The clear cold water rinse shall be performed in accordance with the following:

a. Dip rinsing: A continuous overflow shall be maintained by the addition of fresh water. Items shall be immersed only as long as is necessary to meet the requirements of this specification.

b. The flow of rinse water shall be adjusted, regulated or controlled (e.g. by conductivity controllers) in conjunction with the rate of production in order to prevent its contamination at any time beyond a limit that is such as to assure no detrimental effect on the coatings produced.

c. Spray rinsing may be utilized provided the same end product quality is maintained.

d. Immediately following the water rinse, all items (except class 4) shall be treated in the chronic acid rinse of stage 4 (see 3.2.5).

3.2.5 Stage 4, chromic acid rinse (classes 1, 2 and 3). The chronic acid rinse of classes 1, 2 and 3 shall be performed in accordance with the following:

* a. The final rinse shall be hot 65 to 93 deg. C (150 to 200 deg. F) chronic acid or chromic-phosphoric acid solution: approximately 300 grams chronic acid flake in 1000 liters (L) water.

b. The final rinse shall be maintained at a pH of 2 to 4 by the addition of flake chronic acid or mixtures of chromic and phosphoric acids.

* c. The final rinse shall be checked by a standard free and total acid titration or pH reading as often as is necessary to assure that the bath remains within the limits set at all times during which it is in operation.

* d. All rinses should be discarded whenever they become contaminated. The final rinse shall be checked at least weekly and shall be discarded when the total acid reading rises to more than 7 times the free acid reading.

e. The item should remain in each rinse for a minimum of 60 second(s).

f. Following the chromic acid rinse, the item shall be thoroughly dried before application of a supplementary treatment, as applicable.

* 3.3 Stress Relief. Unless otherwise specified (see 6.2), parts (including carburized parts) having the minimum hardness value of 39 Rockwell C and which are ground, cold formed or cold straightened shall be given a stress relief heat treatment 177 to 204 deg. C (350 to 400 deg. F) for a minimum of one hour(h) for every inch of thickness but not less than half hour before cleaning and coating.

* 3.4 Hydrogen embrittlement relief heat treatment.

3.4.1 Unless otherwise specified (see 6.2), parts (including carburized parts) having the hardness values shown in TABLE I shall be given a hydrogen embrittlement relief beat treatment after coating per TABLE I without any parts developing cracks.

3.4.2 Unless otherwise specified (see 6.2), all lots of parts or material for which baking is required and subject to a sustained load in service exceeding 25 percent of the base metal yield strength shall be tested for hydrogen embrittlement.

* TABLE I. Hydrogen embrittlement heat treatment.

Coating Type	Material	Hardness Rockwell C (minimum)	Heat treatment	Stage of operation
Z	Alloy steel Carbon steel	39 39	98 to 107 deg. C (210 to 225 deg. F) for 8 hours or room temperature for 120 hours	Heat treatment for both types of coatings and material is conducted after coating but before any stressing operation
M	Alloy steel Carbon steel	39 39	98 to 107 deg. C (210 to 225 deg. F) for 8 hours or room temperature for 120 hours	
	Alloy steel Carbon steel	39 39	163 +/- 14 deg. C (325 +/- 25 deg. F) for 4 hours (when approved) (see 6.2)	

* 3.5 Weight of phosphate coatings. The weight of phosphate coatings, prior to application of any supplementary treatment, shall conform to the following.

a. Type M shall be a minimum of 16 grams per square meter (g/m^2) (11 g/m^2 when specified (see 6.2))

b. Type Z shall be a minimum of 11 g/m^2

3.6 Accelerated corrosion resistance. The phosphate coated item, free of supplementary treatment, shall be subjected to a salt spray (fog) test. It shall show no evidence of corrosion for the period of time shown in TABLE II. When authorized by the procuring activity (see 6.2), the Springfield immersion test may be used in lieu of the salt spray test.

3.7 Supplementary treatments. Supplementary treatments shall be applied after completion of the phosphating process in accordance with the following. Items receiving supplementary treatment shall be either centrifuged or permitted to drain sufficiently to remove all surplus from the surfaces.

3.7.1 Types M and Z, class 1. The supplementary treatment for class 1 of types M and Z shall be as specified (see 6.2). Unless otherwise specified, weight of oil per unit area does not apply.

TABLE II. Accelerated corrosion resistance requirement (free of supplementary coating).

Coating system		Exposure time, minimum	
Type	Class	Salt spray, h	Springfield immersion test, minutes
M	1	1-1/2	10
M	2	1-1/2	10
M	3	1-1/2	10
M	4	24	
Z	1	2	10
Z	2	2	10
Z	3	2	10
Z	4	24	

3.7.2 Type M, class 2. The supplementary treatment for class 2 of type M shall be impregnation with lubricating oil conforming to MIL-L-3150 (equivalent to P-7 of MIL-P-116). Unless otherwise specified (see 6.2), the weight of oil per unit area shall be such that items show no signs of corrosion when subjected to the salt spray test for a minimum of 24 hours.

3.7.3 Type M, class 4 systems. Type M, class 4 systems shall be manganese base phosphate coatings that have been chemically converted by reaction with a reagent containing an inorganic salt. When specified (see 6.2), items shall receive supplementary treatment or be dyed to a specific color after chemically converting the phosphate coating. Class 4 coatings shall show no signs of corrosion when subjected to the salt spray test for a minimum of 72 hours only if supplementary treatment is applied.

3.7.4 Type Z, class 2. Type Z, class 2 coatings shall be impregnated with corrosion preventative conforming to grade 1 of MIL-C-16173. When specified (see 6.2), as an alternate method, it shall be permissible to impregnate very small parts with lubricating oil conforming to MIL-L-3150. Unless otherwise approved (see 6.2), the weight per unit area of corrosion preventative shall be such that items shall show no signs of corrosion when subjected to the salt spray test for a minimum of 48 hours.

3.7.5 Type Z, class 4 systems. Type Z, class 4 systems shall be zinc base phosphate coatings which have been chemically converted by reaction with a reagent containing an inorganic salt. When specified (see 6.2), items shall receive supplementary treatment or be dyed to a specific color after chemically converting the phosphate coating. Class 4 coatings shall show no signs of corrosion when subjected to the salt spray test for a minimum of 72 hours only if supplementary treatment is applied.

3.8 Dimensions of coated items. Items shall comply with dimensional requirements of the drawings prior to application of the phosphate coating. After coating, items shall comply with the dimensional requirements of the drawings, with allowance for the phosphate coating buildup. (For interference in close fits, see 6.6).

3.9 Surface texture. Items shall comply with the finish or surface texture requirements of the drawings prior to application of the phosphate coating and after coating when the coating has been stripped as described in 4.8.4.1. Surfaces for which the required characteristics can only be obtained by special mechanical or electro-mechanical operations shall be so specified for proper phosphating. Surface texture, as specified, shall be interpreted in accordance with ANSI B46.1.

3.10 Workmanship. Phosphate coatings shall be evenly deposited, and shall have a uniform crystalline texture with a pattern not readily visible to the unaided eye and shall not produce any evidence of etching or intergranular attack of the base metal. Coatings shall be grey to black, and shall not have a mottled appearance. They shall be free of white stains (due to dried phosphating solution), rust, and fingerprints. However, brown or orange stains caused by chromic acid rinse and non-uniformity of color due to heat treatment, degree of cold work, or composition of the base metal shall not be cause for rejection.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to the prescribed requirements.

* 4.2 Classification of inspection. The inspection requirements specified herein are classified as follows:

a. Preproduction inspection (see 4.5)
b. Quality conformance inspection (see 4.6)

* 4.3 Inspection conditions. Unless otherwise specified (see 6.2), all inspections shall be performed under the following conditions:

a. Temperature: Room ambient 18 to 35 deg. C (65 to 95 deg. F)
b. Altitude: Normal ground
c. Vibration: None
d. Humidity: Room ambient to 95 percent relative, maximum.

* 4.4 Sampling.

4.4.1 Preproduction sample. When specified by the procuring activity (see 6.2), preproduction samples of the item or items to be phosphate coated in accordance with the contract or other similar items approved by the procuring activity shall be coated using the methods and procedures approved in accordance with 3.1. The number of samples shall be as specified in the contract (see 6.2). The sample items shall be subjected to the preproduction inspection detailed in 4.5 at an activity designated by the procuring activity (see 6.2). Acceptance of the preproduction

sample shall be based on no defects in the sample. Further production by the contractor prior to approval of the preproduction sample shall be at the contractor's risk.

* 4.4.2 Lot size. Unless otherwise specified (see 6.2), a lot shall consist of phosphated items of the same base metal composition, type and class of coating, treated under the same conditions, during one shift, in the same processing baths of approximately the same size and shape, and to be submitted for acceptance at one time. In addition, where continuous or conveyor equipment is used, a lot shall consist of a maximum of four consecutive hours of production.

* 4.4.3 Quality conformance inspection sampling.

4.4.3.1 Sampling for examination. Unless otherwise specified (see 6.2), a random sample of coated items shall be taken from each lot in accordance with inspection level II of MIL-STD-105, and subjected to the quality conformance examinations as specified in 4.6.

4.4.3.2 Sampling for tests. Unless otherwise specified (see 6.2), sampling for tests shall be in accordance with the following:

a. Process control tests. Sampling for process control testing shall be as established by the contractor and approved by the procuring activity. The sampling plan shall be sufficient to demonstrate adequate control of the process and conformance of products processed to the requirements of this specification.

b. Quality conformance tests. A random sample of coated items shall be taken from each lot in accordance with inspection level S-1 of MIL-STD-105 and subjected to the quality conformance tests as specified in 4.6.

c. Stress and hydrogen embrittlement relief test. Unless otherwise specified (see 6.2), testing for determination of the adequacy of treatment for the relief of hydrogen embrittlement shall be performed by the processor at a frequency established by bin which shall be demonstrated to assure freedom from failure caused by hydrogen embrittlement by his process for each bath or process line and each type of material processed.

* 4.5 Preproduction inspection. The preproduction inspection shall consist of obtaining approval as specified in 3.1 of procedure, chemicals, and equipment proposed to be used by the contractor for production and, when specified (see 6.2), the tests and examinations specified in TABLE III performed on samples selected in accordance with 4.4.1.

* 4.6 Quality conformance inspection. The quality conformance inspection shall consist of the tests and examinations specified in TABLE III performed on samples selected in accordance with 4.4.3. The stress and hydrogen embrittlememt test shall be performed for each lot only when specified by the procuring activity (see 6.2). Records of bath controls and history shall be maintained by the contractor and made available to the Government upon request as specified in 3.2.1 e and g.

4.7 Examinations.

* 4.7.1 Process examination. The contractor shall allow examination of the phosphate coating process, upon request from the procuring activity, to verify conformance with 3.2.

* 4.7.2 Coated item examination. Samples selected in accordance with 4.4.3.1 shall be examined for compliance with the requirements of 3.8, 3.9 and 3.10. Dimensional and surface roughness measurements after application of the coating need be made only when specified by the procuring activity (see 6.2). The acceptable quality level (AQL) shall be 1.5 percent defective.

4.8 Tests. The AQL for quality conformance tests shall be 4.0 percent defective.

4.8.1 Test specimens. Test specimens shall be in accordance with the following:

a. If the size, shape or cost of the coated item is such that tests cannot reasonably be performed on the item, separate representative test specimens may be utilized. These test specimens shall be supplied by the contractor. Such test specimens shall be cut from scrap items or made from the same alloy, heat treated in the same manner, and surface finished by the same process as the items they represent. The specimens shall have an external surface area of from 10 to 100 square centimeters (cm^2) except as noted in 4.8.3.

b. The test specimens shall be distributed randomly and processed concurrently with the items.

c. The test specimens shall be used only once and then discarded.

* 4.8.2 Process control tests. Process control tests shall be performed at the frequency established in accordance with 4.4.3.2.a. to verify conformance with 3.2.1 through 3.2.5. The contractor’s standard procedures shall be used as approved per 3.1.

TABLE III. Preproduction and quality conformance inspection.

Test or examination	Requirement	Method	Preproduction inspection	Quality conformance inspection
Bath materials and controls	3.2.1	4.7.1 and 4.8.2	X	X
Cleaning	3.2.2	4.7.1 and 4.8.2	X	X
Phosphating	3.2.3	4.7.1 and 4.8.2	X	X
Water rinse	3.2.4	4.7.1 and 4.8.2	X	X
Chromic acid rinse	3.2.5	4.7.1 and 4.8.2	X	X
Stress and hydrogen embrittlement relief	3.3 and 3.4	4.8.3	X	(See note 1)
Weight of coating	3.5	4.8.4	X	X
Accelerated corrosion	3.6	4.8.6	X	X
Supplementary treatments	3.7	4.8.5	X	X
Dimensions of coated items	3.8	4.7.2	X	X
Surface texture	3.9	4.7.2	X	X
Workmanship	3.10	4.7.2	X	X

Notes: 1. The stress and hydrogen embrittlement test shall be performed on each lot only when specified by the procuring activity (see 6.2).

4.8.3 Stress and hydrogen embrittlement relief. Testing for relief of stress and hydrogen embrittlement shall be performed in accordance with the following:

a. Samples of parts that will be subjected to a sustained tensile load in use (see 6.2) shall be subjected to a sustained tensile load equal to 75 percent of the tensile yield strength of the material.

* b. Unless otherwise specified (see 6.2), the parts shall be held under the load for at least 200 hours and then examined visually under 10X magnification and an illumination of 1100 1ux(1x) for cracks. The lot shall be rejected if any coated part develops any crack.

c. Parts which require special fixtures or extreme loads to comply with the above requirements shall be tested as specified by the procuring activity (see 6.2).

* d. For steels having minimum tensile strength of 1655 megapascals (MPa) or higher, cylindrical specimens in accordance with Figure 8 of ASTM E8 with a 0.51 mm notch in the center of the gage length shall be used for test. The area at the base of the notch shall be one-half that of the full diameter of the specimen. The notch shall have an angle of 60 deg.. The radius at the base of the notch shall be 0.254 +/- 0.013 mm for standard 12.8 mm diameter specimens and proportional to this for smaller specimens. These parts shall be subjected to a sustained tensile load equal to 75 percent of the ultimate notch tensile strength of the material.

4.8.4 Weight per unit area of phosphate coatings.

4.8.4.1 Type M coatings. Weight per unit area of phosphate coatings prior to supplementary treatment shall be determined in accordance with the following:

a. Weigh the coated test specimen to the nearest mg; remove the coatings by immersion for a minimum of 15 minutes in a 50 gram per liter (g/L) chromic acid stripping solution at approximately 74 deg. C (165 deg. F); rinse in clean, running water; dry and reweigh.

b. Repeat the procedure until a constant weight is obtained. The chromic acid solution shall not be reused.

* c. If a supplementary treatment is present, it shall be removed prior to testing by immersion in a solvent such as petroleum ether or 1,1,1-trichloroethane. The coating weight shall be calculated as follows:

d. Calculation: Weight per unit area of phosphate coating,

$$W\ (g/m^2) = \frac{\text{(Initial weight in grams-final weight in grams)}}{\text{Total area in square meters}}$$

4.8.4.2 Type Z coatings. Weight per unit area of type Z coatings, prior to supplementary treatment, shall be determined in accordance with the following:

a. Weigh the coated test specimen to the nearest mg; remove the coatings by immersion at room temperature for a minimum of 10 minutes in a stripping solution conforming to TABLE IV. (If desired, the chromic acid stripping solution described in 4.8.4.1a may be used.)

b. Rinse in clean, running water, dry, and reweigh.

c. Repeat the procedure until a constant weight is obtained.

* d. If a supplementary treatment is present, it shall be removed prior to testing as described in 4.8.4.1c. The coating weight shall be calculated as described in 4.8.4.1d.

TABLE IV. Stripping solution for type Z coatings.

Material	Nominal quantity
Sodium hydroxide	180 g
Sodium cyanide	90 g
Water to make	1000 mL

4.8.5 Weight of supplementary treatment. Weight per unit area of supplementary treatment shall be determined in accordance with the following:

a. Age the test samples for a minimum of 16 hours at room temperature, or dry at approximately 51.5 deg. C (125 deg. F) for a minimum of 3 hours. Cool in a desiccator and weigh accurately.

b. Completely remove the supplementary treatment by immersing in successive baths of petroleum ether or naphtha, or by 1,1,1-trichloroethane vapor degreasing.

c. Rinse the sample in alcohol, dry, and reweigh.

d. Determine the weight of the treatment per unit area using the following calculation:

e. Calculation: Weight per unit area of supplementary treatment,

$$W(g/m^2) = \frac{\text{(Initial weight in grams-final weight in grams)}}{\text{area in square meters}}$$

4.8.6 Accelerated corrosion resistance. Accelerated corrosion resistance shall be determined in accordance with 4.8.6.1 or 4.8.6.2 under the conditions specified for each test.

4.8.6.1 Salt spray (fog). Accelerated corrosion resistance by salt spray (fog) shall be performed in accordance with the following:

a. Subject the samples to a 5 percent salt spray test as described in ASTM B 117.

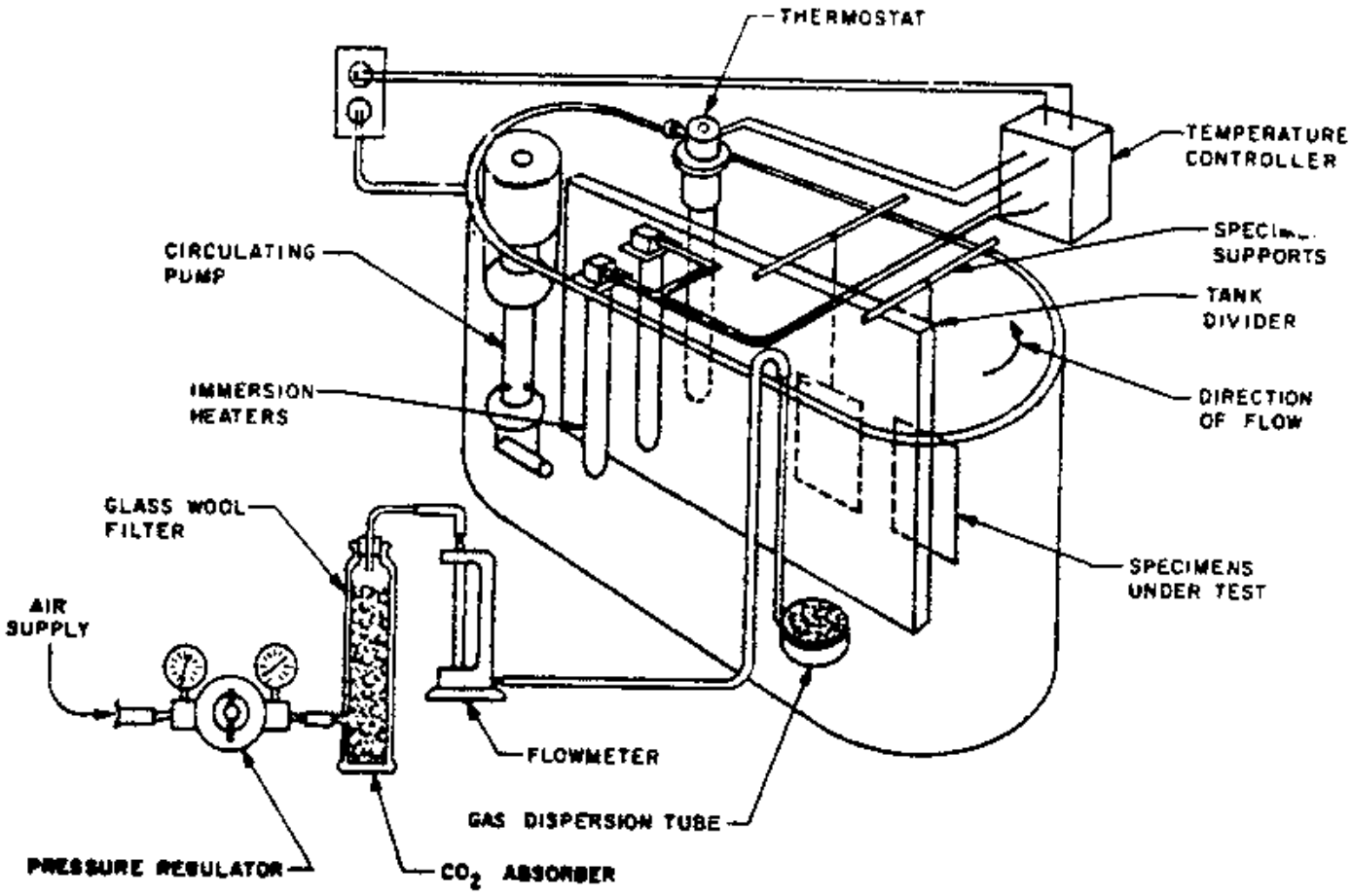
b. Exposure times for coatings tested prior to supplementary treatment shall be in conformance with TABLE II.

c. Exposure time of coatings tested after application of supplementary treatment shall be in accordance with 3.7.

4.8.6.2 Springfield immersion test. If authorized by the procuring activity (see 6.2), the Springfield immersion test may be used in lieu the salt spray test for testing phosphate-coated articles prior to application of a supplementary treatment. The test shall be performed in accordance with the following:

a. Apparatus and conditions: Apparatus capable of providing the proper control of aeration, temperature, velocity, and pH of the solution shall be used to insure adequate reproducibility. See FIGURE 1 for a schematic diagram of typical apparatus suitable for this test.

b. Tank: The tank shall be constructed of material resistant to attack by aerated salt water at elevated temperatures. Glass, fibrous glass-reinforced plastic, or rubber is considered satisfactory for this purpose. The tank shall be of sufficient size to provide at least 1.0L of solution for each 194 cm^2 of specimen surface, and there shall be sufficient additional volume to allow a thermostat sensing element, stirrer, pH meter electrodes, and heating elements to be immersed in the bath. The tank may be rectangular with internally rounded corners (see FIGURE 1), and should be fitted with a lid to facilitate temperature control and to exclude dust and other contaminants.



- FIGURE 1. Schematic diagram of Springfield Immersion Test Apparatus.

c. Aeration, air regulation, and purification: The bath shall be aerated with a regulated flow of air which has been freed of carbon dioxide. The stream of air used to aerate the bath shall be regulated by passage through a pressure regulator and a calibrated flowmeter at the rate of 50 cm[3] per minute per liter of test solution. The regulated stream shall be freed of carbon dioxide by passage through a tower packed with a solid absorbent for CO_2, such as "Ascarite" or soda-lime. The CO_2 absorbing tower shall be connected in the air line between the pressure regulator and the flowmeter (see FIGURE 1). A tower 6.4 cm in diameter and 24 cm in height has been found satisfactory for this purpose; for high rates of air flow, it may be necessary to employ two or more towers connected in parallel. If "Ascarite" is used, the tower shall be recharged with fresh "Ascarite" when the color of the absorbent has changed from white to grey.

d. Air saturation: To obtain air saturation of the test solution, the regulated and purified stream of air shall be blown through a gas dispersion tube having a 20 mm diameter, coarse-fritted glass disk on the end, to break the air stream into small bubbles. Saturation of the bath usually requires approximately one hour. The solution shall be saturated with the air prior to the test, and aeration shall be continued during the test. The degree of saturation may be determined by the Winkler test for dissolved oxygen (see American Public Health Association and American Water Works Association, Standard Methods for the Examination of Water and Waste Water, 13th Edition, 1971).

e. Temperature control: The temperature of the bath shall be maintained at 65 +/- 2 deg. C (149 +/- 3 deg. F) during the test by use of a bimetallic thermo-regulator in conjunction with one or more 500-watt immersion heaters sheathed in fused silica, quartz, glass, or other material resistant to attack by the bath solution.

f. Solution velocity: A fluid velocity of 0.2 m/s shall be maintained in the solution during the test by means of an impeller type pump or a variable-speed zig-zag stirrer. The fluid velocity may be determined by measuring the time required for a float on the surface of the bath to travel a definite distance.

g. pH control: The pH of the solution shall be maintained between 6.8 and 7.0 during the test. The pH may be measured by means of a pH meter having an immersion-type electrode assembly. Dilute sodium hydroxide or hydrocholoric acid may be added to the solution as required to maintain the pH within the specified limits.

h. Specimen supports: The supports for the test specimens shall insulate the specimens from each other and shall allow free contact of the specimens with the test solution. The supports shall be made of a material which will not be attacked by the corroding solution. Glass, plastic laminates and varnished wood have been found to be satisfactory materials for use in supporting specimens.

4.8.6.2.1 Test solution. The test solution shall be in accordance with the following:

a. Preparation: The test solution shall be prepared accurately using sodium chloride conforming to MIL-STD-1218. The solution shall contain 2.0 percent by weight of sodium chloride in freshly boiled distilled water stored in a container made of glass, polyethylene, or other material that will not contaminate water. The opening of the container shall be connected to towers packed with "Ascarite" or soda-lime to protect the water from atmospheric CO_2.

b. Volume: There shall be at least 1.0 L of solution for each 194 cm^2 of specimen surface area to minimize changes in corrosiveness of the solution or accumulation of corrosion products in the bath. The solution shall be discarded after it has been used to test specimens having a combined surface area of 194 cm^2 per liter or after a one-day run if a smaller area has been tested. The volume of the test solution shall be maintained constant by the addition of boiled distilled water to offset any increase in salt concentration.

4.8.6.2.2 Test specimens. The test specimens shall be in accordance with the following:

a. Number of specimens: The number of specimens tested at one time in a bath shall be limited by the ratio of the surface area of specimen to the volume of test solution (see 4.8.6.2.1a).

b. Preparation of specimens: Samples shall be subjected to vapor-phase degreasing with 1,1,1-trichloroethane to free them of oil or grease, rinsed in alcohol, and force-dried at approximately 60 deg. C (140 deg. F).

c. Test procedure: After being immersed in the bath for a minimum of ten minutes under conditions specified in 4.8.6.2, the specimens shall be examined to determine compliance with the requirements of 3.6.

5. PACKAGING

* 5.1 Preservation, packaging, packing and marking. Preservation, packaging, packing and marking for shipment of phosphate coated parts shall be in accordance with the applicable parts specification.

6. NOTES

6.1 Intended use. The phosphate coatings covered by this specification are intended for use where "heavy" coatings are required. Light phosphate coatings used as a paint base are covered by other specifications, such as TT-C-490. However, "heavy" phosphate coatings may be used as a paint base where required on some Naval Gun Systems and Assemblies.

6.1.1 Type M. Type M coatings are more resistant than Type Z to alkaline environments. Type M coatings should not be exposed to temperature in excess of 121 deg. C (250 deg. F). Except for special purpose applications, phosphate coatings should be used with a suitable supplementary treatment. Each class for type M is described below:

a. Class 1 allows the procuring activity to specify a suitable supplementary treatment depending upon the particular application.

b. Class 2 covers coatings impregnated with MIL-L-3150 lubricating oil. This class may be specified for general purpose applications in which a moderate degree of corrosion resistance is required and to prevent wear and assist in the "breaking-in" of bearing surfaces. MIL-L-3150 oil may be removed from phosphate coated items by contact with absorbing material.

c. Class 3 covers coatings without supplementary treatment. This class is intended for special purposes in which a supplementary treatment is not desired.

d. Class 4 covers chemically converted coatings, chemically converted with supplementary treatments that are suitable for use where corrosion protection is required in addition to other properties (see 1.2). This coating is recommended for use where a dyed or colored coating such as olive drab or a black (non-reflective) coating is needed. The class 4 coating provides improved corrosion resistance; however, the supplementary coating provides most of the corrosion protection when used in conjunction with phosphate coatings. The class 4 coating provides an improved "break-in" coating and is recommended for reducing the sliding friction in preference to the class 2. The use of the class 4 coating reduces the variation in torque versus tension values usually found with threaded fasteners.

6.1.2 Type Z. Type Z coatings should not be used if contact with alkaline materials or exposure to temperatures above 93 deg. C (200 deg. F) is expected. Type Z coatings may be used to prevent galling in cold-extrusion and deep-drawing applications. Except for special-purpose applications, Type Z coatings should be used with a suitable supplementary treatment (see 1.2). Each class for type Z is described below:

a. Class 1 allows the procuring activity to specify a suitable treatment depending upon the particular application.

b. Class 2 covers coatings impregnated with grade 1 of MIL-C-16173. This class may be specified for general purpose application in which corrosion protection is the primary consideration and where a "dry-to touch" film is required, indoor or outdoor, for domestic and overseas shipment, with or without cover. MIL-C-16173, grade 1 compound is equivalent to P-1 of MIL-P-116.

c. Class 3 covers coatings without supplementary treatment. This class is intended for special purposes in which a supplementary treatment is not desired.

d. Class 4 covers chemically converted coatings with supplementary treatments that are suitable for use where petroleum base or wax supplementary treatments cannot be used but where extended corrosion protection is necessary. The color of the chemically converted coatings will be grey (see 1.2). Class 4 chemically converted coatings, treated with a high melting wax or a preservative oil, provide excellent corrosion protection where the wax or oil is not objectionable. This coating is recommended for use where a dyed or colored coating such as olive drab or a black (non-reflective) coating is needed. The class 4 coating provides improved corrosion resistance; however, the supplementary coating provides most of the corrosion protection when used in conjunction with phosphate coatings. The class 4 coating provides an improved "break-in" coating and is recommended for reducing the sliding friction in preference to the class 2. The use of the class 4 coating reduces the variation in torque versus tension values usually found with threaded fasteners.

6.2 Ordering data. Procurement documents should specify the following:

* 6.2.1 Procurement requirements.

a. Title, number and date of this specification.

b. Type and class of coating (see 1.2).

c. When preproduction inspection is not required (see 3.1 and 6.3).

d. When phosphate coating (and any supplementary treatment) is not to be the final process (see 3.2).

e. When frequency of bath sampling is not the contractor’s option (see 3.2.1f).

f. When abrasive/blasting is not required as pretreatment (see 3.2.2).

g. When stress and hydrogen embrittlement relief is not required (see 3.3 and 3.4).

h. When type M coating weight shall be 11 g/m$^{[2]}$ (see 3.5).

i. When Springfield immersion test is allowed (see 3.6 and 4.8.6.2).

j. Supplementary treatment required for class 1 and weight of treatment (see 3.7.1).

k. When weight of supplementary treatment for class 2 is other than as specified (see 3.7.2 and 3.7.4).

1. Supplementary treatment and color of dye desired for type M or type Z, class 4 (see 3.7.3 and 3.7.5), when applicable.

m. Inspection conditions if other than as specified (see 4.3).

n. Assigned activity for preproduction inspection (see 4.4.1).

o. Number and type of preproduction samples required.

p. Specify if any exception to lot size and sampling plans (see 4.4.2 and 4.4.3.1).

q. Then stress and hydrogen embrittlememt relief tests are required for each lot; frequency if not for each lot; exceptions and details of procedure (see 4.4.3.2c, 4.6, TABLE III and 4.8.3).

r. When tests and examinations are required for preproduction inspection (see 4.5).

s. When surface roughness and dimensional measurements are required after coating (see 4.7.2).

* 6.2.2 Contract data requirements. Any data required for delivery in connection with this document shall be specified on a DD Form 1423 incorporated into the contract. Such data will be delivered as identified on completed (numbered) DIDs (Data Item Descriptions/DD 1664 which will be documented in the Applicable ADL (Authorized Data List).

* 6.3 Preproduction sample waiver. Preproduction samples submitted and approved on a recent contract may be accepted by the procuring activity in lieu of an additional preproduction sample inspection. When the preproduction sample is waived (see 6.2.1c), the procurement document should contain a statement specifying that the standards of workmanship exhibited by the previously approved preproduction sample shall determine the minimum requirements of the current contract or order.

6.4 Supersession data. TABLE V shows the classification comparison of this specification with coatings specified in previous editions of MIL-P-16232 and MIL-C-12968.

6.4.1 Phosphating materials. Phosphating solutions previously included in MIL-P-16232 are now covered in MIL-P-50002. Phosphating materials complying with MIL-P-50002 should be procured and used by Government agencies to produce coatings conforming to MIL-P-16232.

* 6.5 Surface finishes finer than 0.80 micrometers. Abrasive blasting using too coarse a media grit size may destroy surface finishes less

than 0.80 micrometers. The grit size must be selected to be compatible with the surface finish requirement and it may be necessary to use 320 grit size or grade 1000 glass beads. Phosphate coatings should not be specified for highly polished surfaces that cannot tolerate fine vapor abrasive blasting. Phosphate coating is not recommended for surfaces of 0.20 micrometers or less (see 3.2.2.1d).

TABLE V. Classification comparison.

DOD-P-16232F & MIL-P-16232E	MIL-P-16232D	MIL-P-16232C	MIL-P-16232B	MIL-C-16232A	MIL-C-12968
Type M, Class 1	Type M, Class 1	Type M, Class 1	Type M, Class 1	-	Type A
Type M, Class 2	Type M, Class 2	Type M, Class 2	Type M, Class 2	Type I	-
Type M, Class 3	Type M, Class 3	Type M, Class 3	Type M, Class 3	-	-
Type M, Class 4	Type M, Class 4A	Type M, Class 4A	-	-	-
New Class 4 deleted 4A,B,C,& D	Type M, Class 4B	Type M, Class 4B	-	-	-
	Type M, Class 4C	-	-	-	-
	Type M, Class 4D	-	-	-	-
	Type M, Class 4E	-	-	-	-
Type Z, Class 1	Type Z, Class 1	Type Z, Class 1	Type Z, Class 1	-	Type B, Class 1
Type Z, Class 2[1]	Type Z, Class 2[1]	Type Z, Class 2[2]	Type Z, Class 2[3]	Type II	-
Type Z, Class 3	Type Z, Class 3	Type Z, Class 3	Type Z, Class 3	-	-
Type Z, Class 4	Type Z, Class 4A	Type Z, Class 4A	Type Z, Class 4	-	Type B, Class 2
New Class 4 deleted 4A,B,C, & D	Type Z, Class 4B	Type Z, Class 4B	-	-	-
	Type Z, Class 4C	Type Z, Class 4C	-	-	-
	Type Z, Class 4D	Type Z, Class 4D	-	-	-
	Type Z, Class 4E	Type Z, Class 4E	-	-	-

[1] Treated with MIL-C-16173, Grade 1 (or MIL-L-3150 for very small parts).
[2] Treated with MIL-C-16173, Grade 2.
[3] Treated with MIL-C-16173, Grade 1A.

6.6 Dimensions of coated items. TABLE VI gives expected thickness of phosphate coatings. If close fits are specified, buildup in thickness caused by the phosphate coating may result in apparent interference on assembly. Coatings are friable, however, and assembly may be accomplished by forced fitting, wiping, or otherwise removing the surplus coating. Nonferrous metal wire brushes should not be employed. Salt spray or immersion test requirements are applicable to coated articles prior to removal of any coating in assembly operations.

TABLE VI. Expected thickness of phosphate coatings.

Type	Thickness ([mu]m)
M	5 to 10
Z	5 to 15

* 6.7 Changes from previous issue. The margins of this specification are marked with an asterisk to indicate where changes (additions, modifications, corrections, deletions) from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

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